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Full Paper

Electrochemical, Thermodynamic, and Surfaces Investigations on The Use of *Mentha Piperita* Essential Oil as A Green Corrosion Inhibitor for Carbon Steel in 1 M HCl Solution

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Abstract- The inhibiting impact of *Mentha piperita* essential oil utilized as a natural corrosion preventer for Carbon steel in 1 M hydrochloric acid solution was examined utilizing electrochemical impedance spectroscopy (EIS) and potentiodynamic polarization (PDP) methods, UV-visible spectra and scanning electron microscopy (SEM)/(EDS). The results of these examinations demonstrated that *Mentha piperita* essential oil was a significant inhibitor. The inhibition efficacy (%IE) improved with rising essential oil of *Mentha piperita* leaves concentration and decreasing temperature. At 100 ppm, the extract showed exceptional inhibitory action, with an efficiency of 89.6% at 303 K. According to polarization curves, the *Mentha piperita* essential oil functions as a mixed inhibitor, it was clarified by the carbon steel surface absorbing *Mentha piperita* essential oil. The adsorption process follows the Langmuir isotherm. Studies on electrochemical impedance spectroscopy revealed a single capacitive loop, demonstrating the procedure of a charge transfer was responsible for controlling the corrosion reaction. The kinetic activation and adsorption process' thermodynamic characteristics were also estimated and explained.

Keywords- *Mentha piperita* essential oil; Carbon steel corrosion; EIS/PDP; Adsorption; UV-visible/SEM/EDS

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1. INTRODUCTION

Carbon steel is used extensively throughout a variety of industries because of its adaptability, low cost, and simple production [1,2]. Nevertheless, corrosion can also affect carbon steel. which during many applications can be a serious problem. Corrosion is the term used to describe the gradual degradation of a metal caused by contact with the environment, notably the presence of humidity and oxygen [3,4]. Corrosion can cause structural damage, reduce an item's usable life, and increase maintenance costs [5]. Inhibitors of corrosion are added to acidic solutions to reduce the rate at which metals corrode [6]. Metals corrode more quickly in acidic liquids because hydrogen ions are present there. The corrosion inhibitors stop hydrogen ions from getting into an interface with the metal by creating a barrier on its surface. The development of the protective layer could be caused by the production of a passive oxygen layer or by the molecules of the inhibitors adhering to the metal surface .

The constituents of essential oils, aromatic plant extracts, have been discovered to offer several advantageous qualities, including the capacity to act as corrosion inhibitors [7-9].

Several advantages come with using essential oils as corrosion inhibitors, including their natural origin, low toxicity, and biodegradability [10–12]. Additionally, essential oils are more widely available and useful than traditional synthetic corrosion inhibitors.

Owing to the existence of aromatic bioactive components and terpenoids, *Mentha piperita* is used to treat conditions such as ulcers, colds, headaches, cancer, cough, irritation, inflammation, diabetes, and digestive system issues [13].

Studies have shown that essential oils like tea tree oil [14], cinnamon oil [15], citrus oil [16], and thyme oil [17] can effectively prevent carbon steel from corroding. These essential oils contain compounds with known anticorrosive effects [18–20] such as eugenol, cinnamaldehyde, limonene and thymol, and linalool. For essential oils to operate as corrosion inhibitors, a layer of defence must form on the metal surface. Essential oils and the metallic ions on the surface come combined to form a substance that acts as a barrier to stop further corrosion.

In the current study, the effectiveness of inhibition and kinetic of the *Mentha piperita* leaves essential oil to prevent carbon steel from corroding in a 1 M HCl solution were examined using polarization potentiodynamics, EIS, UV-visible analysis, and surface characterization of the carbon steel using scanning electron microscopy (SEM) combined with energy dispersive spectroscopy (EDS), before and after the film formation.

2. EXPERIMENTAL TECHNIQUES

2.1. Isolation of essential oil

Mentha piperita leaves were stored at room temperature for 14 days in a dry place without light. Essential oils were isolated by hydrodistillation using a Clevenger apparatus method. Dried leaves were placed in a 250 mL Erlenmeyer flask, distilled water was added until the leaves were completely covered, and then distillation was done for 3 hours. The essential oils were then stored at 4 °C until further analysis.

2.2. Material

Coupons made of carbon steel with the following composition (Table 1) were utilized for the corrosion testing as a working electrode:

Table 1. Steel elemental composition expressed as a mass percentage

Element	C	Si	Mn	S	Cr	Ti	Ni	Co	Cu	Fe
wt%	0.370	0.230	0.680	0.016	0.077	0.011	0.059	0.009	0.160	Balance

In order to get trustworthy and reproducible results, the working electrode was pre-treated before each experiment by polishing the electrode surface with increasingly fine abrasive paper (SiC 180, 400, 1500, and 1200), then rinsing using distilled water and drying.

2.3. Solution

The acidic solution (1 M HCl) was created using diluting concentrated acid with distilled water, which had a density of 1.19 and a purity of 37%. The hydrochloric acid content remained at 1 M as a result. The needed concentrations of the inhibitor were dissolved in an acidic solution, and the uninhibited solution was provided as a reference to compare.

Before each experiment, *Mentha piperita* essential oil was added directly to the corrosive solution to create test solutions. There are four various concentrations of *Mentha piperita* essential oil: 100 ppm, 75 ppm, 50 ppm, and 25 ppm. To ensure reproducibility, three examples were used in the tests.

2.4. Electrochemical techniques

The electrochemical experiment was performed with a Potentiostat PGZ 100 in a typical electrolytic cell with a 3-electrode with Volta-Master software control. The reference electrode was a saturated calomel electrode (SCE), a platinum electrode as a counter electrode, and the material used to construct the working electrode was carbon steel. The exposed electrode surface to the corrosive electrolyte is 1 cm^2. At a sweep rate of 0.5 mV/s, potentiodynamic polarization curves were plotted. 30 min were given for the electrode potential to stabilize before the measurements began. At 303 K, polarization curves were measured between -800 and 0 mV vs. SCE.

The effectiveness of the inhibition was evaluated using values i_{corr} determined using the relation given below:

$$\eta_{PDP} = \left(1 - \frac{i'_{corr}}{i^0_{corr}}\right) \times 100 \qquad (1)$$

where i^0_{corr} and i'_{corr} are, respectively, the corrosion currents without and with *Mentha piperita* essential Oil.

In the frequency range of 100 kHz to 10 mHz with an amplitude of 10 mV, electrochemical impedance measurements were made.

The inhibition effectiveness got from the polarization resistance is evaluated employing the following relation:

$$\eta_{EIS} = \left(1 - \frac{R^0_P}{R'_P}\right) \times 100 \qquad (2)$$

where R^0_P and R'_P are the resistance of polarization without and in with inhibitor, respectively.

In the absence of an inhibitor, we exploited the results already published by our team for both stationary and transient polarization techniques for the effect of temperature and concentration, since we worked under the same conditions [21].

2.5. UV-vis measurement

UV-visible spectra were employed in this work on the corrosive solution before and after submerging the carbon steel surface in 1 M HCl for 72 hours at 303 K in the presence of the optimal concentration of *Mentha piperita* essential Oil, UV-vis studies were performed on the surface. UV-visible spectrum Analysis was carried out using the Jasco V-730 spectrophotometer.

2.6. Scanning electron microscope

A 24-hour immersion period with and without the inhibitors examined at their optimum concentration was monitored by use of a scanning electron microscope (SEM) to gather data on the adsorption behavior of *Mentha piperita* essential oil on the carbon steel surface. To check the composition of the material, an X-ray dispersion (EDS) device connected to an electron balance microscope was used.

3. RESULTS AND DISCUSSION

3.1. Concentration impact

3.1.1. PDP curves

The kinetics of metal corrosion is still studied via experiments on polarization curves[21]. The polarization curves of carbon steel in the acid solution with and without *Mentha piperita* essential oil as presented in Figure 1. When the *Mentha piperita* essential oil is introduced, all of the cathode and anode branches move down, as can be seen. Additionally, this phenomenon makes more sense as *Mentha piperita* essential oil concentration increases. Although the shape of the cathodic branches has not changed much, the cathodic reaction mechanism has not changed. This is explained by the fact that molecules of *Mentha piperita* essential oil adhere to metal surfaces, inhibiting active sites and lessening metal corrosion as a result [22,23].

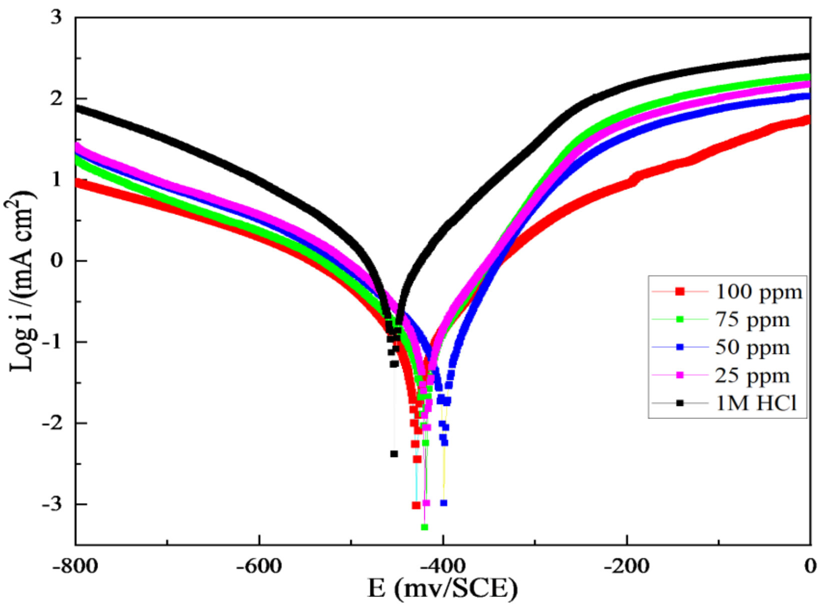

Figure 1. Tafel polarization curves for carbon steel at different *Mentha piperita* essential oil concentrations in 1 M HCl at 303K

Table 2. Tafel polarization data obtained at different concentrations of *Mentha piperita* essential oil on carbon steel in 1 M HCl at 303 K

Conc.	$-E_{corr}$ (mV/ SCE)	i_{corr} (μA cm^{-2})	β_a (mV dec^{-1})	$-\beta_c$ (mV dec^{-1})	η_{PDP} (%)
1 M	456.3	1104.1	112.8	155.4	---
25 ppm	418.2	194.2	81.8	130.3	82.4
50 ppm	399.0	182.5	73.9	157.7	83.4
75 ppm	420.1	169.7	78.8	154.0	84.6
100 ppm	428.4	122.2	103.7	127.9	88.9

Table 2 displays the essential kinetic parameters including inhibitory effectiveness (η_{PDP}%), cathodic and anodic Tafel slopes (β_c and β_a), corrosion potential (E_{corr}), and corrosion current density (i_{corr}).

The polarization parameters obtained employing the Tafel extrapolation technique are shown in Table 2. According to this table, when *Mentha piperita* essential oil is added, the high i_{corr} value in the initial solution is reduced. This attitude is brought on by the molecules of *Mentha piperita* adhering to the carbon steel surface. the formation of a barrier layer prevents harsh ions from directly attacking the surface of carbon steel by obstructing the active cathodic and anodic sites of the metal surface [24]. As a result, the adsorbed coating slows down the dissolving of the metal in the acidic environment. According to a published study, the material may be categorized as anodic/cathodic if the E_{corr} voltage drop is at least 85 mV in comparison to the blank solution [25,26]. Table 1 data shows that the *Mentha piperita* essential oil in the under-evaluation method acted as an inhibitor of a mixed kind. These outcomes confirm the theory that the *Mentha piperita* essential oil under investigation greatly reduced the moderate cathodic reaction and the anodic dissolving response.

3.1.2. EIS study

The Nyquist plots of carbon steel in 1 M HCl solution at 303 K with and without *Mentha piperita* essential oil are shown in Figure 2. One semi-cercle can be seen in all of the obtained impedance spectra, proving that the transfer of charge and corrosion are related processes [27,28]. The presence of *Mentha piperita* essential oil the diameter of the semi-cercles is considerably larger than that seen in the blank, which may be related to the formation of a defensive layer on the surface of the carbon steel [29].

Figure 3 demonstrates the charge transfer control of corrosion reactions, revealing the existence of a unique time constant. The rise in phase angle (close to -90°) indicates that the deposited protective layer has stronger barrier properties and more capacitive behavior [30]. The phase angle of Mentha piperita-treated samples is superior to that of blank, particularly under optimal conditions. The existence of a unique time constant in the Bode and Nyquist diagrams demonstrated the protective mechanism is charge transfer. For all experimental impedance data, the following model provided an excellent fit ($\chi \leq 10^{-3}$) [31].

The conventional equivalent circuit seen in Figure 4 was fitted to the EIS data. The values of the constant phase element (CPE), which reflects the capacitance of the electric double layer (C_{dl}) and adsorption inhibitor film, R_s the resistance of the solution, R_P the resistance for polarization, and CPE for the adsorption inhibitor film**.**

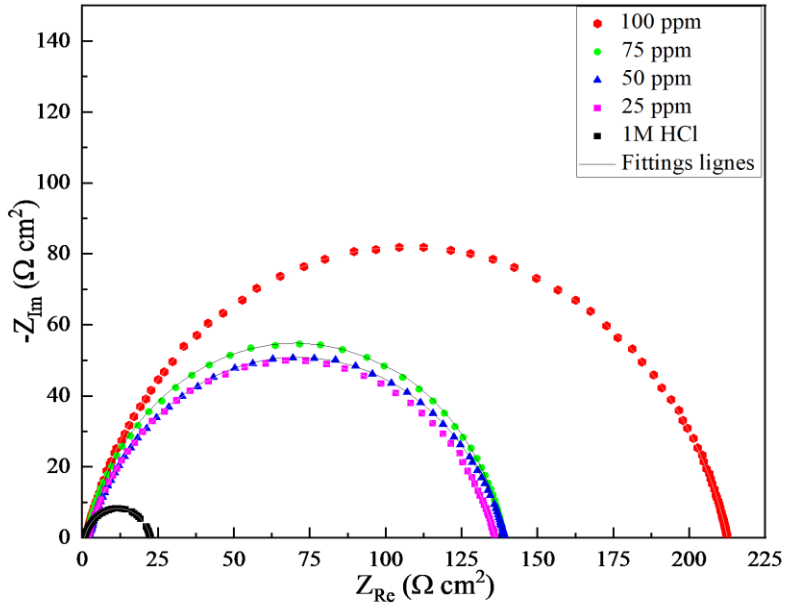

Figure 2. Nyquist plots of carbon steel in 1 M HCl containing different concentrations of Mentha piperita essential oil at 303 K

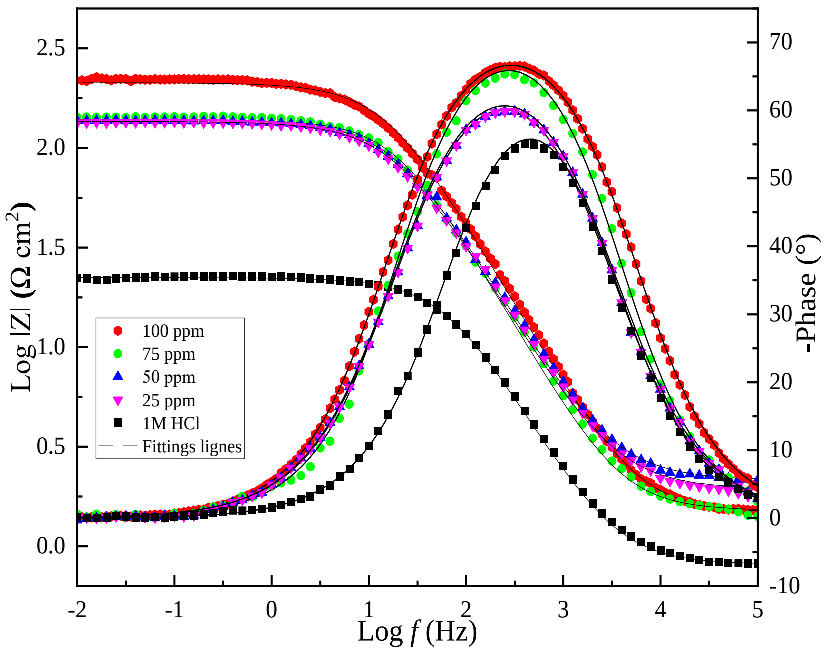

Figure 3. Bode graphs for carbon steel at 303 K in 1 M HCl without and with different Mentha piperita essential oil concentrations at 303 K

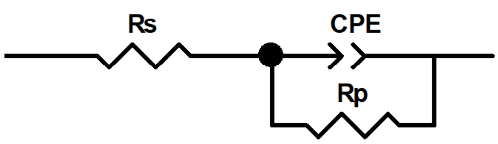

Figure 4. Equivalent circuit for EIS data fitting

Table 3. EIS data of carbon steel at 303 K in 1 M HCl without and with different Mentha piperita essential oil concentrations

C	R_s (Ω cm^2)	R_p (Ω cm^2)	$10^6\times A$ (μF s^{n-1} /cm^2)	n	C_{dl} (μF /cm^2)	χ^2	η_{EIS} (%)
1 M	0.83	21.57	293.9	0.845	116.2	0.002	---
25 ppm	2.49	128	128.3	0.847	61.1	0.006	83.2
50 ppm	2.82	130.9	124.9	0.847	59.4	0.008	83.5
75 ppm	2.26	134	120.0	0.851	58.2	0.006	83.9
100 ppm	1.52	208.4	89.6	0.854	45.3	0.009	89.6

As can be seen in Table 3, impedance measurements were used to calculate the double layer capacitance (C_{dl}) and resistance of charge transfer (R_p), and these values were then used to compute the inhibitory efficiency.

The C_{dl} values are calculated by using the following formula [32]:

$$c_{dl} = \left(AR_p^{(1-n)}\right)^{\frac{1}{n}} \qquad (3)$$

Furthermore, we conclude that as inhibitor concentration increases, R_p values also rise, increasing inhibition efficiency to 89.6% at 100 ppm. In fact, a charge-transfer process that primarily affects carbon steel corrosion is confirmed by the presence of Mentha piperita essential oil, which is accompanied by an increase in the R_p value in 1 M HCl. When the inhibitor is present, double-layer capacitance values are also reduced to their lowest possible level. The decrease in C_{dl} is caused by the inhibitor's adhesion onto the metal surface, which forms a layer or complex on the surface of carbon steel in 1 M HCl solution [33].

3.1.3. Isotherm for adsorption

The adsorption isotherms are extremely common and frequently employed to investigate the different forms of interactions and to offer fundamental understandings of the adsorption processes (physically, chemically, or both) in order to offer important knowledge on the adsorption of the chemical substances that serve as inhibitors. The Langmuir adsorption isotherm was evaluated in the present investigation to identify the most appropriate adsorption isotherm, and it was discovered to fit our experimental findings adequately (Figure 4) [34,35]. The equation below can be used to express this isotherm:

$$\frac{C_{inh}}{\theta} = \frac{1}{K_{ads}} + C_{inh} \qquad (4)$$

where θ the recovery rate, C_{inh} is the inhibitor concentration and K_{ads} is the process's equilibrium constant for adsorption.

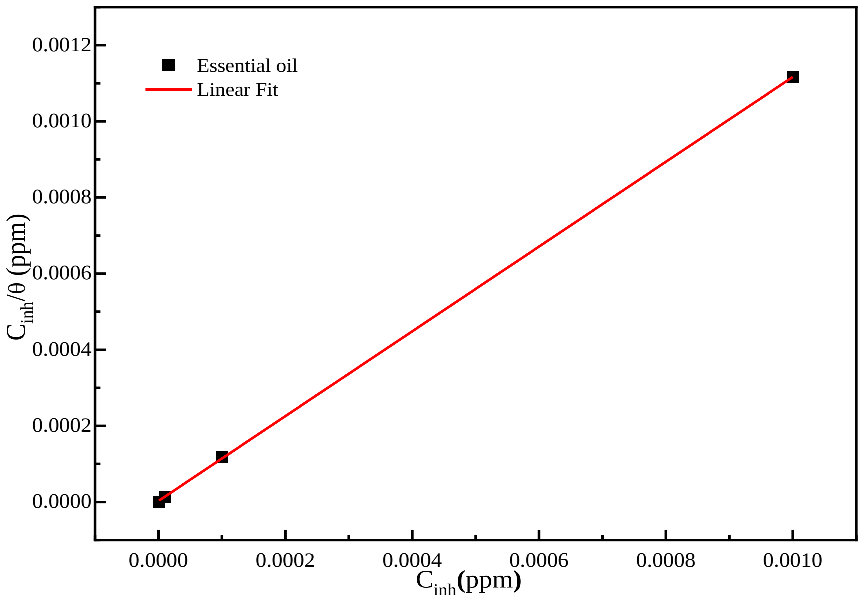

Figure 5. Langmuir adsorption of Mentha piperita essential oil in a 1 M HCl solution on carbon steel at 303 K

Table 4. Thermodynamic data of the adsorption of Mentha piperita essential oil in 1 M HCl on carbon steel at 303 K

	K_{ads} (ppm^{-1})	Slope	R^2
Mentha piperita essential oil	363591.4	1.114	0.999

Figure 5 demonstrates the straight line that results from the relationship between C_{inh}/θ and C_{inh} at 303 K, with a correlation coefficient R^2 that is nearly equivalent to one ≈0.999 and a slope that is also almost one. Furthermore, a strong relationship between the adsorbate and the surface is indicated by a high K_{ads} value (Table 4). This behavior shows that the adsorption of Mentha piperita essential oil on a carbon metal surface follows the Langmuir adsorption isotherm. Furthermore, it is very important to highlight that because the molecular masses of the extract constituents are unknown, it is not viable to describe the adsorption isotherm behavior when utilizing natural product extracts as inhibitors in terms of the conventional free energy of adsorption value [36].

3.2. Temperature impact

The importance of studying temperature impact on the corrosion of stainless steel in acidic solutions cannot be overstated [37]. In this study, we varied the temperature between 303 and 333 K to see how this element would impact Mentha piperita essential oil ability of inhibition with and without the addition of 100 ppm of inhibitor (Figure 6). In addition, it is possible to calculate the thermodynamic and chemical parameters, which enables the identification of the adsorption mechanism type.

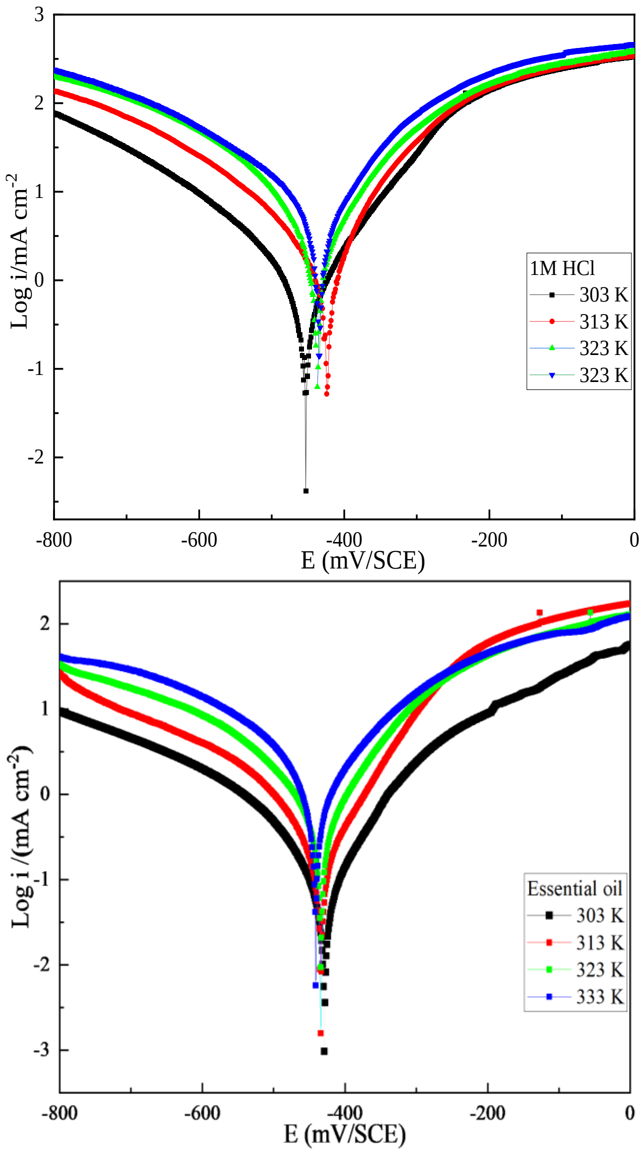

Figure 6. PDP curves for carbon steel in 1 M HCl at different temperatures without and with the addition of 100 ppm Mentha piperita essential oil

Discovering how temperature impacts an inhibitor's capacity to prevent corrosion may help us better understand how corrosion is inhibited because the majority of chemical processes are known to be affected by temperature. PDP experiments are used in this case to explore how temperature affects corrosion rate. at temperatures that range from 303 to 333 K. The outcomes are shown in Table 4. It has been demonstrated that raising the temperature hastens the disintegration of the metal in both environments with and without 100 ppm Mentha piperita essential oil. However, compared to uncontrolled solutions, corrosion is significantly slowed down when the optimum oil concentration is added to 1 M HCl, and the values of η_{PDP} were increased with rising temperature from 303 to 333 K. Regardless of the presence or absence of 100 ppm Mentha piperita essential oil in solution, the i_{corr} values increased as the temperature climbed, showing that steel corrosion was accelerated up by the higher temperature. The corrosion inhibitor slows down the speed of corrosion when it is introduced to the solution, by adhering to the metal's surface and forming a barrier.

Table 5. PDP parameters and corrosion inhibiting effectiveness of carbon steel in 1 M HCl without and with the addition of 100 ppm *Mentha piperita* essential oil at different temperatures

Medium	T (K)	$-E_{corr}$ (mV/SCE)	i_{corr} (μA/cm²)	β_a (mV /dec)	$-\beta_c$ (mV /dec)	η_{PDP} (%)
Blank	303	456.3	1104.1	112.8	155.4	-----
	313	423.5	1477.4	91.3	131.3	-----
	323	436.3	2254	91.4	117.8	-----
	333	433.3	3944.9	103.9	134.6	----
Mentha piperita	303	428.39	122.22	103.7	127.9	88.9
	313	433.02	202.08	82.7	102.4	86.3
	323	433.61	394.44	84.8	94.7	82.5
	333	440.70	1001.8	117.6	110.9	74.6

3.3. Thermodynamic analysis

Because various changes, including quick etching, inhibitor desorption, and possible disintegration of the inhibitor, occur on the metal surface, the temperature effect on the inhibited acid-metal reaction is quite complicated. The activation thermodynamic parameters of the corrosion process were determined utilizing the Arrhenius Equation below [38]:

$$\ln(i_{corr}) = \ln A - \frac{E_a}{RT} \quad (4)$$

$$\ln\left(\frac{i_{corr}}{T}\right) = \ln\frac{R}{Nh} + \frac{\Delta S_a}{R} - \frac{\Delta H_a}{RT} \quad (5)$$

where A defines the pre-exponential constant (Arrhenius constant), E_a the energy of activation, h defines Plank's constant, N expresses Avogadro's number, ΔH_a the enthalpy, and ΔS_a the entropy.

The Arrhenius traces of ln i_{corr} as a function of 1/T and ln i_{corr}/T as a function of 1/T are shown in Figure 7&8. She displays straight lines with regression coefficients that are extremely near to 1 to demonstrate that the corrosion process being investigated conforms with the Arrhenius equation with a slope of ($-E_a$ / R). Table 6 contains the computed and measured activation energy (E_a). Considering the evaluation of the data in Table 6. Displays that the apparent activation energy of the inhibitor is lower than that of the uninhibited solution, which was explained by the physical adsorption of Mentha piperita essential oil onto the surface of carbon steel [39]. In contrast to the blank solution, the value of ΔH_a was raised in the presence of inhibitors, showing a higher level of protective efficiency and better protective effectiveness in contrast to the uninhibited solution. This might be because there is an energy barrier to the reaction, which causes the enthalpy of the corrosion process to increase during the process of the inhibitor's adsorption [40].

The negative significance of ΔS_a in both without and with Mentha piperita essential oil suggests that formation activation complex indicates an association step rather than a dissociation step. The presence of Mentha piperita essential oil led to a positive increase in activation entropy compared to the absence of an inhibitor. This suggests that as reactants transform into activated complexes, there is a decrease in disorder. However, the rise in ΔS_a suggests that disordering increases when one transitions from the response to the activation compound [41].

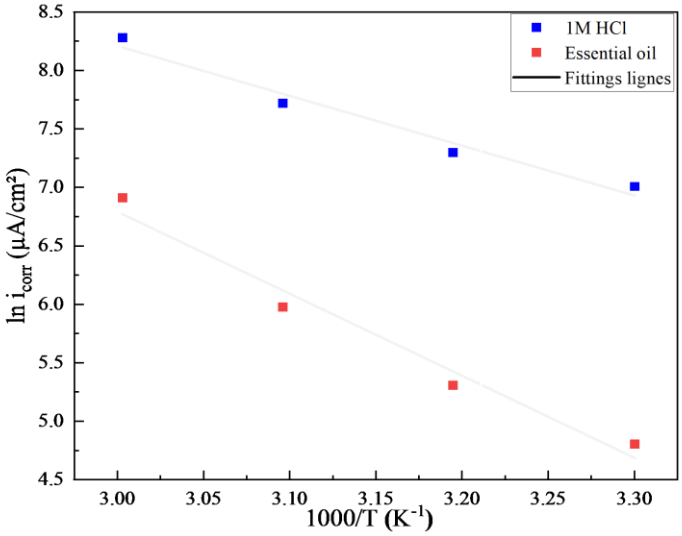

Figure 7. Arrhenius lines of carbon steel in 1 M HCl medium in the absence and presence of Mentha piperita essential oil

Table 6. Activation parameters for dissolving carbon steel in 1 M HCl without and with the presence of Mentha piperita essential oil

Medium	E_a (kJ mol^{-1})	ΔH_a (kJ mol^{-1})	ΔS_a (J mol^{-1}K^{-1})
HCl 1 M	35.4	32.76	-79.18
Mentha piperita essential oil	58.26	55.6	-22.39

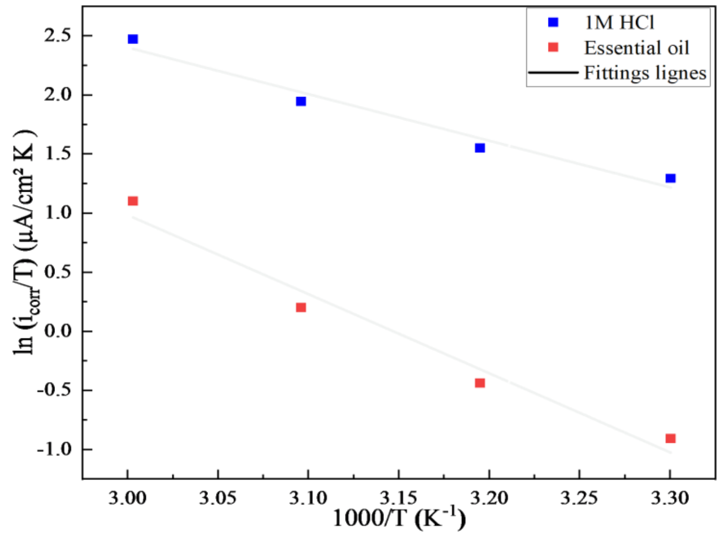

Figure 8. Arrhenius lines of ln i_{cor}/T=f(1/T) of carbon steel in 1 M HCl medium in the absence and in the presence of Mentha piperita essential oil

3.4. UV-visible spectra

To support the probable creation of a complex Mentha piperita-Fe, Figure 9 shows the spectra of UV-visible absorption measured before and after carbon steel was submerged in 1 M HCl solution containing 100 ppm of *Mentha piperita* essential oil for 3 days. The electronic bands of compound solutions (Figure 9) clearly exhibit bands of absorption at 231.2 nm for *Mentha piperita* essential oil before immersing the carbon steel in a 1 M solution of HCl.

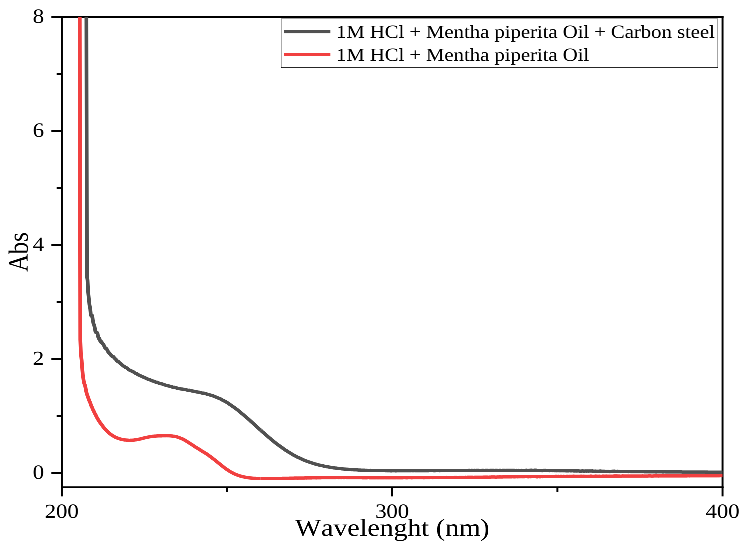

Figure 9. UV-visible spectra of the 1 M HCl solution in the presence of *Mentha piperita* essential oil before and after 24 h immersion in carbon steel

This band has a high charge transfer characteristic and is linked to the transition π-π* involving the entire electronic structure of *Mentha piperita* essential oil. However, the highest absorption bands exhibited a bathochrome shift from 231.2 to 246.4 nm after the carbon steel was three days submerged in an aggressive solution with 100 ppm of *Mentha piperita* essential oil. A shift in the location of the maximum absorption, or λ_{max}, in accordance with the research gathered, denotes the development of a complex between the dispersed species. The requisite proof that a compound between Fe^{2+} and *Mentha piperita* essential oil may form in a fondued HCl 1 M is provided by the experimental results we performed [42,43].

3.5. Scanning electron microscopy

Figure 10 displays the SEM images of the carbon steel surface before immersion (Figure 10 (a)) with and without the addition of 100 ppm of *Mentha piperita* essential oil, after being submerged in HCl 1 M solutions for 24 hours. In this study, we used the SEM-EDS results in the absence of the inhibitor from a work already published by our research team [45].

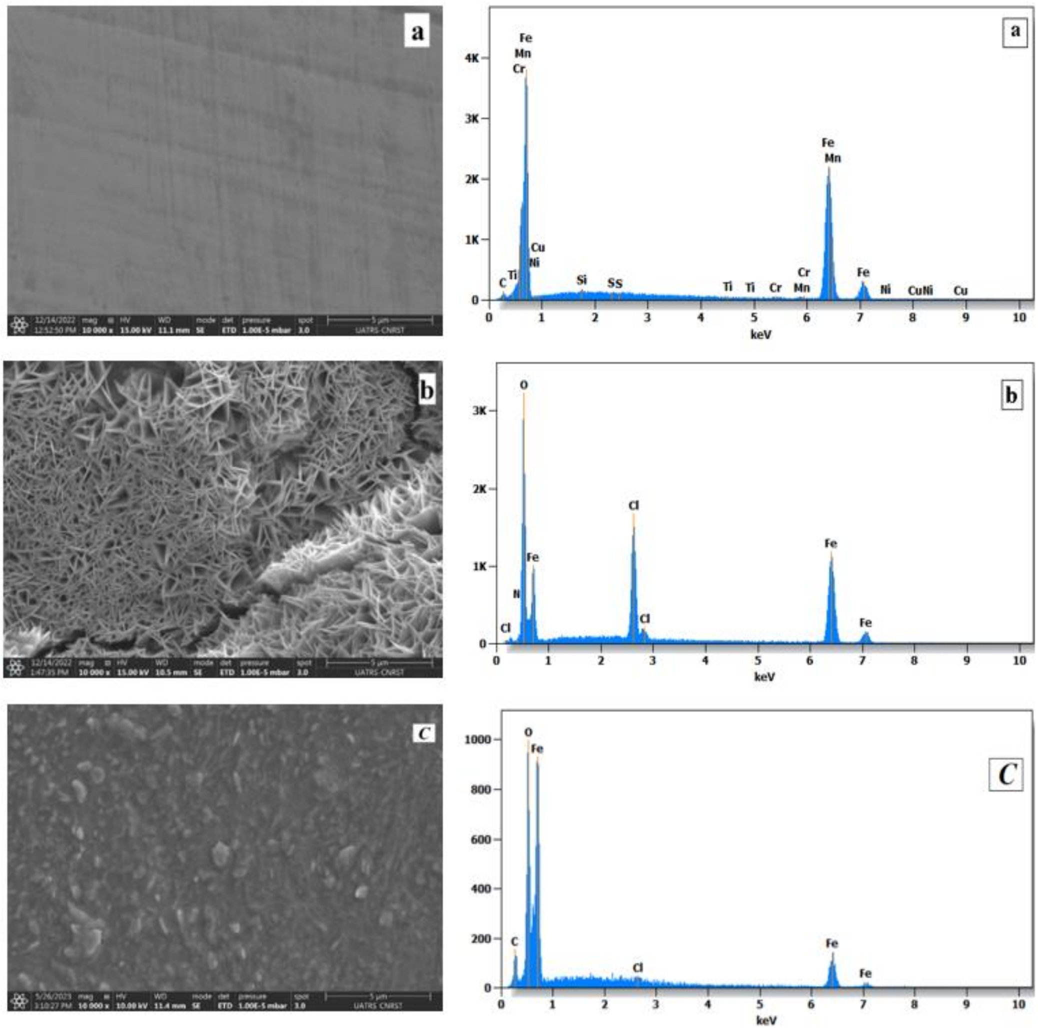

Figure 10. SEM/EDS analysis of carbon steel specimens (a) before immersion, (b) after 24 h immersion in 1M HCl and (c) 1M HCl + 100ppm of *Mentha piperita* essential oil at 303 K

It is clear from Figure 10 (b) that the HCl solution badly damaged the metal's surface; this damage may have been caused by the excessive amount of the metal's alloy that was dissolved.

Numerous cracks and dispersed pits are apparent. However, the Carbon steel's surface greatly improved, and a film that inhibits rusting developed with the addition of 100 ppm of *Mentha piperita* essential oil to the corrosive environment (Figure 10(c), which decreased metal dissolution and offered good corrosion protection. The electrochemical data covered in the previous section are supported by the organic material traces visible on the SEM images. The EDS's findings examining the chemical properties of the layer that has developed on the surface of carbon steel (Figure 10) These findings demonstrate the formation of a heteroatom film at the surface that contains O and is considered to have active centers for the adsorption process. The production of corrosion products including Cl and/or the adsorption of these ions onto the metal's surface are two possible ways that Cl might manifest.

4. CONCLUSION

This study has evaluated the effectiveness of *Mentha piperita* leaves as a natural corrosion inhibitor utilizing electrochemical methods, UV-vis, and surface investigation. In a 1 M HCl solution, the extract exhibited good carbon steel inhibition characteristics. The efficacy of the inhibition lowered when the temperature rises and the concentration of the inhibitor increases. The outcomes from potentiodynamic polarization, electrochemical impedance spectroscopy (EIS) techniques, and surface characterization. The extract demonstrated outstanding inhibitory performance at 100 ppm, with an efficiency of 89.6% at 303 K. The SEM investigation. shown that the inhibitor adsorption forms a barrier on the carbon steel's surface. It was found that the adsorption of the extracted film followed the Langmuir isotherm.

Declarations of interest

The authors declare no conflict of interest in this reported work.

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